

This Page Is Inserted by IFW Operations
and is not a part of the Official Record

BEST AVAILABLE IMAGES

Defective images within this document are accurate representations of the original documents submitted by the applicant.

Defects in the images may include (but are not limited to):

- BLACK BORDERS
- TEXT CUT OFF AT TOP, BOTTOM OR SIDES
- FADED TEXT
- ILLEGIBLE TEXT
- SKEWED/SLANTED IMAGES
- COLORED PHOTOS
- BLACK OR VERY BLACK AND WHITE DARK PHOTOS
- GRAY SCALE DOCUMENTS

IMAGES ARE BEST AVAILABLE COPY.

**As rescanning documents *will not* correct images,
please do not report the images to the
Image Problem Mailbox.**



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification ⁵: A61K 7/06, 7/00	A1	(11) International Publication Number: WO 94/12148 (43) International Publication Date: 9 June 1994 (09.06.94)
(21) International Application Number: PCT/US93/10940 (22) International Filing Date: 15 November 1993 (15.11.93) (30) Priority Data: 983,337 30 November 1992 (30.11.92) US (71) Applicant: EASTMAN KODAK COMPANY [US/US]; 343 State Street, Rochester, NY 14650 (US). (72) Inventors: MYERS, Gary, Lynn; 908 Colfax, Kingsport, TN 37665 (US). HILLER, John, Jacob; 5332 Foxfire Place, Kingsport, TN 37664 (US). MINGA, Robin, Lynn; 193 Oak Street, Blountville, TN 37617 (US). DOBBS, Suzanne, Winegar; 304 Highridge Road, Kingsport, TN 37660 (US). SINGLETON, Andy, Hugh; 453 Eastley Court, Apartment 4, Kingsport, TN 37660 (US). (74) Agent: THALLEMER, John, D.; 343 State Street, Rochester, NY 14650-2201 (US).		(81) Designated States: CA, JP, KR, European patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published <i>With international search report. Before the expiration of the time limit for amending the claims and to be republished in the event of the receipt of amendments.</i>
(54) Title: AEROSOL HAIR SPRAY FORMULATIONS (57) Abstract This invention relates to aerosol hair spray formulations based on (1) a sulfonate-containing, water-dispersible or water-dissipatable, linear polyester having a glass transition temperature of 36 °C to 40 °C and (2) a water-soluble, polyvinyl lactam polymer. In addition, the formulations contain water as the liquid vehicle and a propellant. Such aerosol hair spray formulations do not contain any volatile organic compounds other than propellant yet exhibit fast drying times and excellent performance characteristics. <div style="text-align: right; margin-top: 100px;"><i>Myers et al.</i></div>		

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

AT	Austria	GB	United Kingdom	MR	Mauritania
AU	Australia	GE	Georgia	MW	Malawi
BB	Barbados	GN	Guinea	NE	Niger
BE	Belgium	GR	Greece	NL	Netherlands
BF	Burkina Faso	HU	Hungary	NO	Norway
BG	Bulgaria	IE	Ireland	NZ	New Zealand
BJ	Benin	IT	Italy	PL	Poland
BR	Brazil	JP	Japan	PT	Portugal
BY	Belarus	KE	Kenya	RO	Romania
CA	Canada	KG	Kyrgyzstan	RU	Russian Federation
CF	Central African Republic	KP	Democratic People's Republic of Korea	SD	Sudan
CG	Congo	KR	Republic of Korea	SE	Sweden
CH	Switzerland	KZ	Kazakhstan	SI	Slovenia
CI	Côte d'Ivoire	LI	Liechtenstein	SK	Slovakia
CM	Cameroon	LK	Sri Lanka	SN	Senegal
CN	China	LU	Luxembourg	TD	Chad
CS	Czechoslovakia	LV	Latvia	TG	Togo
CZ	Czech Republic	MC	Monaco	TJ	Tajikistan
DE	Germany	MD	Republic of Moldova	TT	Trinidad and Tobago
DK	Denmark	MG	Madagascar	UA	Ukraine
ES	Spain	ML	Mali	US	United States of America
FI	Finland	MN	Mongolia	UZ	Uzbekistan
FR	France			VN	Viet Nam
GA	Gabon				

AEROSOL HAIR SPRAY FORMULATIONS

FIELD OF THE INVENTION

This invention relates to aerosol hair spray
5 formulations based on (1) a sulfonate-containing,
water-dispersible or water-dissipatable, linear
polyester having a glass transition temperature of 36°C.
to 40°C. and (2) a water-soluble, polyvinyl lactam
polymer. In addition, the formulations contain water as
10 the liquid vehicle and a propellant. Such aerosol hair
spray formulations do not contain any volatile organic
compounds other than propellant yet exhibit fast drying
times and excellent performance characteristics.

BACKGROUND OF THE INVENTION

15 Hair spray formulations typically comprise a
solution of a polymer, the fixative, in water/alcohol
mixtures. The polymeric materials which are typically
used in hair spray formulations are soluble in water or
20 water/alcohol mixtures and are derived from N-vinyl-
pyrrolidinone or N-vinylpyrrolidinone and one or more
other vinyl monomers such as vinyl acetate, acrylate and
methacrylate esters and/or styrene compounds. When
applied to hair and allowed to dry, the polymeric
25 material provides human hair body, consistency, firm
texture, and, in general, maintains the hair in a
desired arrangement.

Significant amounts of volatile organic compounds
such as alcohols are present in such hair spray
30 formulations to facilitate rapid drying of the polymer
solution. Environmental concerns continue to encourage
the development of hair spray formulations which contain
very little and preferably no volatile organic
compounds. Attempts to omit the volatile organic
35 component of hair sprays have failed to produce

formulations which have acceptable drying times, particularly when the water level exceeds 55% by weight of the formulation.

U.S. Pat. No. 4,300,580 describes hair spray
5 formulations containing a water-dispersible or
water-dissipatable linear sulfo-polyester fixative in a
water/alcohol mixture. Such formulations are fast
drying and have good hair holding properties but possess
the disadvantage of being very difficult to remove from
10 the hair. For example, prolonged washing is required to
completely remove the water-dispersible, linear
polyester fixative to obtain hair with no tacky or
sticky feel. In an effort to overcome the fixative
removal problem, U.S. Pat. No. 4,300,580 teaches the
15 addition of certain water soluble polymers to
formulations containing the water-dispersible, linear
polyester. The use of poly(alkylene glycols) such as
poly(ethylene glycol) is disclosed. However, when such
formulations containing a combination of the
20 poly(alkylene glycol) and water-dispersible, linear
polyester are applied to hair and allowed to dry, the
fixative causes a matting of the hair. Such matting
hinders combing, brushing and styling of hair. It is
important to note that U.S. Pat. No. 4,300,580
25 recommends in column 4, lines 36-38, a "nonaerosol"
method of application of the hair spray formulation.

U.S. Pat. No. 4,150,216 discloses grooming
formulations containing branched sulfo-polyesters.
Difficulty, however, is encountered in maintaining the
30 specified molecular weight range of 600 to 5,000.
Slight variations in the condensation temperatures
and/or times results in branched polymers having
molecular weights which exceed the desired values and
which have poor film forming characteristics, i.e. they
35 are hard and brittle, and are not readily water

dispersible or soluble as required in hair spray formulations.

The present inventors have unexpectedly discovered that aerosol hair spray formulations wherein the
5 fixative is a combination of two polymeric materials:
(1) a sulfonate-containing, water-dissipatable, linear polyester having a Tg of 36°C. to 40°C. and (2) a water-soluble, polyvinyl lactam polymer, exhibit improved properties over aerosol hair sprays containing
10 a sulfo-polyester having a Tg outside of said range. The aerosol hair spray formulations of the present invention do not have the disadvantages described hereinabove, such as tackiness, matting and difficulty in removal. Moreover, the aerosol hair spray
15 formulations do not contain any volatile organic compounds other than propellant.

SUMMARY OF THE INVENTION

Accordingly, it is one object of the present
20 invention to provide an aerosol hair spray formulation having improvements in one or more of the above desirable features.

Accordingly, it is another object of the invention to provide an aerosol hair spray formulation which is
25 not tacky, has a fast drying rate, acceptable body, consistency and firm texture necessary to hold hair in the desired arrangement for a certain length of time and does not contain any volatile organic compounds.

Still another object of the invention is to provide
30 an aerosol hair spray formulation having excellent storage stability and which does not clog or produce foam at the exit port of an aerosol container.

These and other objects are accomplished herein by an aerosol hair spray composition comprising:

35 (1) 1 to 10 weight percent based on the weight of

components (1), (2), (3) and (4) of a sulfo-polyester having a glass transition temperature of 36°C. to 40°C. consisting essentially of repeat units from

- 5 (a) a dicarboxylic acid selected from the group consisting of aromatic dicarboxylic acids, saturated aliphatic dicarboxylic acids, cycloaliphatic dicarboxylic acids, and combinations thereof;
- (b) a diol; and
- 10 (c) a difunctional sulfomonomer containing at least one sulfonate group attached to an aromatic nucleus wherein the functional groups are hydroxy, carboxy or amino, provided the difunctional sulfomonomer is
- 15 present in an amount from 4 to 25 mole percent based on 100 mole percent dicarboxylic acid and 100 mole percent diol; and

(2) 1 to 7 weight percent based on the weight of components (1), (2), (3) and (4) of a water-soluble, polyvinyl lactam polymer containing at least 50 mole percent of residues of N-vinyl lactams of the formula



wherein n is 3 or 4;

(3) 46 to 94 weight percent based on the weight of components (1), (2), (3) and (4) of a liquid vehicle consisting essentially of water; and

(4) 3 to 40 weight percent based on the weight of components (1), (2), (3) and (4) of a propellant selected from the group consisting of a

40 C₁ - C₄ aliphatic hydrocarbon, dimethyl ether, and mixtures thereof.

DESCRIPTION OF THE INVENTION

The term "hair" as used in the present invention includes treated and untreated human hair, animal hair, and any type of fiber which requires consistency and firm texture necessary to hold it in the desired arrangement for a certain length of time.

The sulfo-polyester, component (1), has a glass transition temperature in the critical range of 36°C. to 40°C. and contains repeat units from a dicarboxylic acid, a diol and a difunctional sulfomonomer. Dicarboxylic acids useful in the present invention include aromatic dicarboxylic acids preferably having 8 to 14 carbon atoms, saturated aliphatic dicarboxylic acids preferably having 4 to 12 carbon atoms, and cycloaliphatic dicarboxylic acids preferably having 8 to 12 carbon atoms. Specific examples of dicarboxylic acids are: terephthalic acid, phthalic acid, isophthalic acid, naphthalene-2,6-dicarboxylic acid, cyclohexanedicarboxylic acid, cyclohexanediacetic acid, diphenyl-4,4'-dicarboxylic acid, succinic acid, glutaric acid, adipic acid, azelaic acid, sebacic acid, and the like. The polyester may be prepared from two or more of the above dicarboxylic acids.

It should be understood that use of the corresponding acid anhydrides, esters, and acid chlorides of these acids is included in the term "dicarboxylic acid".

The diol component of the polyester includes cycloaliphatic diols preferably having 6 to 20 carbon atoms or aliphatic diols preferably having 3 to 20 carbon atoms. Examples of such diols are: ethylene glycol, diethylene glycol, triethylene glycol, 1,4-cyclohexanedimethanol, propane-1,3-diol, butane-1,4-diol, pentane-1,5-diol, hexane-1,6-diol, 3-methylpentanediol-(2,4), 2-methylpentanediol-(1,4),

2,2,4-trimethylpentane-diol-(1,3),
2-ethylhexanediol-(1,3), 2,2-diethylpropane-diol-(1,3),
hexanediol-(1,3), 1,4-di-(hydroxyethoxy)-benzene,
2,2-bis-(4-hydroxycyclohexyl)-propane,
5 2,4-dihydroxy-1,1,3,3-tetramethyl-cyclobutane,
2,2-bis-(3-hydroxyethoxyphenyl)-propane, and
2,2-bis-(4-hydroxypropoxyphenyl)-propane. The polyester
may be prepared from two or more of the above diols.

The difunctional sulfomonomer component of the
10 polyester may be a dicarboxylic acid or an ester thereof
containing a sulfonate group ($-SO_3^-$), a diol containing
15 a sulfonate group, or a hydroxy acid containing a
sulfonate group. The cation of the sulfonate salt may
be Na^+ , Li^+ , K^+ , NH_4^+ , and substituted ammonium. The
20 term "substituted ammonium" refers to ammonium
substituted with an alkyl or hydroxy alkyl radical
having 1 to 4 carbon atoms. The difunctional
sulfomonomer contains at least one sulfonate group
25 attached to an aromatic nucleus wherein the functional
groups are hydroxy, carboxy or amino. Advantageous
difunctional sulfomonomer components are those wherein
the sulfonate salt group is attached to an aromatic acid
nucleus such as benzene, naphthalene, diphenyl,
30 oxydiphenyl, sulfonyldiphenyl or methylenediphenyl
nucleus. Preferred results are obtained through the use
of sulfophthalic acid, sulfoterephthalic acid,
sulfoisophthalic acid, 4-sulfonaphthalene-2,7-
dicarboxylic acid, and their esters. The sulfomonomer
35 is present in an amount from 4 to 25 mole percent,
preferably 10 to 12 mole percent, based on 100 mole
percent dicarboxylic acid and 100 mole percent diol.

In particularly preferred embodiments, the water
dispersible sulfo-containing linear polyester is derived
40 from (a) a mixture of dicarboxylic acids consisting of
isophthalic acid (or ester) and 5-sodio-sulfoisophthalic

acid, (b) a diol component consisting of diethylene glycol, or a mixture of diols consisting of at least 75 mole percent of diethylene glycol with the remaining diol being either ethylene glycol or

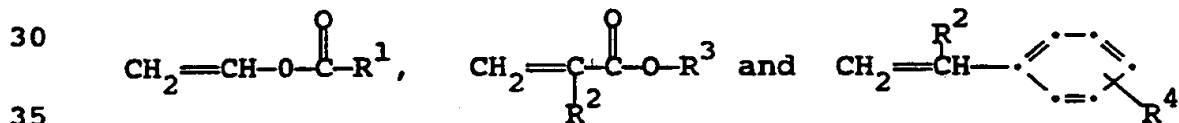
5 1,4-cyclohexanedimethanol. The sulfo-polyester, component (1), is present in an amount of 1 to 10 weight percent, based on the weights of components (1), (2), (3) and (4) of the aerosol hair spray formulation.

10 Component (2) is a water soluble vinyl polymer or copolymer which contains at least 50 mole percent of the residues of n-vinyl lactam monomer the formula



20

In the above formula, n is 3 or 4. Preferably, n equals three wherein the N-vinyl lactam monomer is N-vinylpyrrolidinone. Examples of other vinyl compounds which may be copolymerized with an N-vinyl lactam to 25 prepare the water-soluble polymers include vinyl esters and vinyl aromatic compounds having the structure



40 wherein R^1 is alkyl, e.g., straight- and branched-chain alkyl of up to 10 carbon atoms; R^2 is hydrogen or
45 methyl; R^3 is alkyl, e.g., straight- and branched-chain alkyl of up to 10 carbon atoms, and alkyl substituted by
50 hydroxy or by amino including alkylamino and dialkylamino; and R^4 is hydrogen or alkyl of up to 4 carbon atoms.

55 The water-soluble polymers, component (2), may be

prepared according to known procedures wherein a N-vinyl lactam is polymerized, optionally in the presence of one or more other vinyl monomers such as those described above. The N-vinylpyrrolidinone/vinyl acetate
5 copolymers supplied by BASF under the trademark LUVISKOL VA are typical of the water-soluble polymers which may be used in the aerosol hair spray compositions of the present invention. The preferred water-soluble polymers comprise homopolymers of N-vinyl-2-pyrrolidinone and
10 copolymers of N-vinyl-2-pyrrolidinone and up to 50 mole percent vinyl acetate having weight average molecular weights in the range of 1000 to 100,000. The water-soluble, polyvinyl lactam polymer, component (2), is present in an amount of 1 to 7 weight percent, based
15 on the weights of components (1), (2), (3) and (4) of the aerosol hair spray formulation.

Component (3) of the aerosol hair spray formulations is a liquid vehicle. The liquid vehicle of the formulations may be water or a water/alcohol
20 mixture. Distilled or deionized water are the preferred sources of water since tap water generally contains ions which would precipitate the sulfopolyester, component (1). The alcohol should have two to four carbon atoms. Specific alcohols include, ethanol,
25 isopropanol and t-butanol. A preferred water/alcohol mixture contains 55 to 65 weight percent water and 35 to 45 weight percent alcohol. The preferred alcohol is ethanol. The liquid vehicle is present in an amount of 46 to 94 weight percent, based on the weights of
30 components (1), (2), (3) and (4) of the aerosol hair spray formulation.

Component 4 is a propellant selected from the group consisting of a $C_1 - C_4$ aliphatic hydrocarbons and
35 dimethyl ether. The aliphatic hydrocarbons may be branched or straight chain and include methane, ethane,

propane, n-butane, isobutane, or mixtures thereof. A preferred aliphatic hydrocarbon propellant is a mixture containing 83 percent isobutane and 17 percent propane. The propellant is present in an amount of 3 to 40 weight percent, based on the weights of components (1), (2), (3) and (4) of the aerosol hair spray formulation. In the case where a $C_1 - C_4$ aliphatic hydrocarbon is used as the propellant, generally 3 to 10 weight percent, preferably 4 to 7 weight percent, is employed. In the case where dimethyl ether is used as the propellant, generally, 30 to 40 weight percent, preferably, 30 to 35 weight percent, is employed.

Other conventional additives such as preservatives, fragrances, antifoaming agents, hair conditioners, plasticizers, etc. may be added in such quantities as desired, up to 5.0% by weight of the total formulation. Although the film-forming formulations described herein are particularly useful as aerosol hair sprays for the grooming of hair, it is possible that the formulations, with or without modification, may be used in other types of personal care products.

It is unexpected, based on the prior art, that the combination of a sulfo-containing water dispersible polyester having a T_g of 36°C. to 40°C. with a water soluble vinyl polymer would give aerosol hair spray formulation improvements over either of the single component systems or a dual component system at other glass transition temperatures, particularly in washability/rinsability, tackiness, humidity resistance and film elasticity.

The materials and testing procedures used for the results shown herein are as follows:

DYMEL A (trademark) (CTFA Adopted Name: Dimethyl Ether) available from DuPont, is a dimethyl ether and is used as a propellant.

LUVISKOL VA 73W PVP/VA (CTFA Adopted Name: PVP/VA Copolymer), available from BASF, is a water soluble vinyl copolymer of 70 mole percent of N-vinyl-2-pyrrolidinone and 30 mole percent of vinyl acetate (50% solids), and is used as a fixative.

GLYDANT (trademark) (CTFA Adopted Name: DMDM Hydantoin) available from Lonza, Inc. is 1-(hydroxymethyl)5,5-dimethyl hydantoin, and is used as a antimicrobial.

Inherent viscosity (I.V.) was measured at 23°C. using 0.50 grams of polymer per 100 ml of a solvent consisting of 60% by weight phenol and 40% by weight tetrachloroethane.

Preparation of tresses involved natural brown, European virgin hair. two grams of hair, root end, were glued to a 2" by 2" (5.1 cm x 5.1 cm) plastic tab. The tresses were cut so that the length of hair hanging below the tabs was six inches (15.2 cm)

The invention will be further illustrated by a consideration of the following examples, which are intended to be exemplary of the invention. All parts and percentages in the examples are on a weight basis unless otherwise stated.

EXAMPLE I

Preparation of a water-dispersible sulfo-polyester

A 500 mL round bottom flask equipped with a ground-glass head, an agitator shaft, nitrogen inlet and a side arm was charged with 74.0 grams of isophthalic acid, 16.0 grams of 5-sodiosulfoisophthalic acid, 106.0 grams of diethylene glycol, sufficient titanium isopropoxide to provide 50 ppm of titanium, and 0.45 grams of sodium acetate tetrahydrate. The flask was immersed in a Belmont bath at 200°C. for two hours under a nitrogen sweep. The temperature of the bath was

increased to 280°C. and the flask was heated for one hour under reduced pressure of 0.5 to 0.1 mm of Hg. The flask was allowed to cool to room temperature and the copolyester was removed from the flask. The copolyester had an I.V. of 0.45 and a glass transition temperature of 30°C. as measured using a differential scanning calorimeter (DSC). The copolyester was extruded and pelletized.

10

EXAMPLE II

Preparation of an aerosol hair spray formulation

The copolyester prepared in Example I, 7.14 grams, was dispersed in 52.15 grams of distilled water by heating and stirring until a temperature of 85°C. was reached. After cooling to 40°C. any water lost during heating was replaced and 5.71 grams of a water soluble vinyl copolymer consisting of 70 mole percent of N-vinyl-2-pyrrolidinone and 30 mole percent of vinyl acetate (50% solids) was added at 40°C. The mixture was stirred and filtered to remove any residual material. The pH was adjusted to 5.5 +/- 0.5 followed by the addition of 35.0 grams of ethanol.

To 61.88 grams of the composition was added 15.4 grams of a mixture containing 83 percent isobutane and 17 percent propane. The formulation was applied in the form of an aerosol hair spray to hair tresses. The test results are summarized in Table I.

EXAMPLE III

Preparation of a water-dispersible sulfo-polyester

A 500 mL round bottom flask equipped with a ground-glass head, an agitator shaft, nitrogen inlet and a side arm was charged with 74.0 grams of isophthalic acid, 16.0 grams of 5-sodiosulfoisophthalic acid, 83.0 grams of diethylene glycol, 16.0 grams of

1,4-cyclohexanedimethanol, sufficient titanium isopropoxide to provide 50 ppm of titanium, and 0.45 grams of sodium acetate tetrahydrate. The flask was immersed in a Belmont bath at 200°C. for one hour under a nitrogen sweep. The temperature of the bath was increased to 230°C. for one hour. The temperature of the bath was increased to 280°C. and the flask was heated for 45 minutes under reduced pressure of 0.5 to 0.1 mm of Hg. The flask was allowed to cool to room temperature and the copolyester was removed from the flask. The copolyester had an I.V. of 0.36 and a glass transition temperature of 38°C. as measured using a differential scanning calorimeter (DSC). The copolyester was extruded and pelletized.

EXAMPLE IV

Preparation of an aerosol hair spray formulation

The copolyester prepared in Example III, 17.85 grams, was dispersed in 217.9 grams of distilled water by heating and stirring at 80-85°C. for 15 minutes. The mixture was cooled to 40°C. Water lost during heating was replaced by adding distilled water. A water soluble vinyl copolymer consisting of 70 mole percent of N-vinyl-2-pyrrolidinone and 30 mole percent of vinyl acetate (50% solids), 14.3 grams, was added and the mixture was stirred and filtered to remove residual material. The pH was adjusted to 5.5 +/- 0.5 and 0.2 weight percent of 1-(hydroxymethyl)-5,5-dimethyl hydantoin was added. Good storage stability of the formulation was observed after aging at 40°C. for one week in an oven.

To 70.0 grams of the above hair spray formulation was added 30.0 grams of dimethyl ether. The all aqueous aerosol formulation showed good clarity and storage stability. The formulation was applied in the form of

an aerosol hair spray to hair tresses. The test results are summarized in Table I.

EXAMPLE V

5 Preparation of an aerosol hair spray formulation

The copolyester prepared in Example III, 7.14 grams, was dispersed in 52.15 grams of distilled water by heating and stirring until a temperature of 85°C. was reached. After cooling to 40°C. any water lost during
10 heating was replaced and 5.71 grams of a water soluble vinyl copolymer consisting of 70 mole percent of N-vinyl-2-pyrrolidinone and 30 mole percent of vinyl acetate (50% solids) was added at 40°C. The mixture was stirred and filtered to remove any residual material.
15 The pH was adjusted to 5.5 +/- 0.5 followed by the addition of 35.0 grams of ethanol.

To 61.88 grams of the composition was added 15.4 grams of a mixture containing 83 percent isobutane and 17 percent propane. The formulation was applied in the
20 form of an aerosol hair spray to hair tresses. The test results are summarized in Table I.

EXAMPLE VI

Preparation of a water-dispersible sulfo-polyester

25 A 500 mL round bottom flask equipped with a ground-glass head, an agitator shaft, nitrogen inlet and a side arm was charged with 136.0 grams of isophthalic acid, 53.0 grams of 5-sodiosulfoisophthalic acid, 155.0 grams of diethylene glycol, 78.0 grams of
30 1,4-cyclohexanedimethanol, sufficient titanium isopropoxide to provide 50 ppm of titanium, and 1.48 grams of sodium acetate tetrahydrate. The flask was immersed in a Belmont bath at 200°C. for one hour under a nitrogen sweep. The temperature of the bath was
35 increased to 230°C. for one hour. The temperature of

the bath was increased to 280°C. and the flask was heated for 45 minutes under reduced pressure of 0.5 to 0.1 mm of Hg. The flask was allowed to cool to room temperature and the copolyester was removed from the flask. The copolyester had an I.V. of 0.33 and a glass transition temperature of 55°C. as measured using a differential scanning calorimeter (DSC). The copolyester was extruded and pelletized.

10

EXAMPLE VII

Preparation of an aerosol hair spray formulation

The copolyester prepared in Example VI, 17.85 grams, was dispersed in 217.9 grams of distilled water by heating and stirring at 80-85°C. for 15 minutes. The mixture was cooled to 40°C. Water lost during heating was replaced by adding distilled water. A water soluble vinyl copolymer consisting of 70 mole percent of N-vinyl-2-pyrrolidinone and 30 mole percent of vinyl acetate (50% solids), 14.3 grams, was added and the mixture was stirred and filtered to remove residual material. The pH was adjusted to 5.5 +/- 0.5 and 0.2 weight percent of 1-(hydroxymethyl)-5,5-dimethyl hydantoin was added. Good storage stability of the formulation was observed after aging at 40°C. for one week in an oven.

To 70.0 grams of the above hair spray formulation was added 30.0 grams of dimethyl ether. The sulfo-polyester precipitated. Dimethyl ether was replaced with ethane and the sulfo-polyester precipitated. Thus, the sulfo-polyester having a Tg of 55°C. was not compatible with any of the propellants which are necessary in aerosol hair spray formulations.

EXAMPLE VIII

In order to evaluate the effect of the glass transition temperature of the linear sulfo-polyester in a aerosol hair spray formulation, tresses were treated with the aerosol hair spray formulations of Examples II and V. The aerosol hair spray formulation of Example II contains a sulfo-polyester having a Tg of 30°C. while the aerosol hair spray formulation of Example V contains a sulfo-polyester having a Tg of 38°C. other than the glass transition temperature, the formulations were essentially identical. One tress per treatment was used. The tresses were sprayed with the respective aerosol hair spray for 10 seconds each which was enough time to completely cover each of the tresses. Individual tresses were dried under heat while subjective feel tests were conducted on the wet tresses. The subjective evaluations were conducted by a panel of ten people. The evaluators rated the tresses on a scale of 1 to 10. The lower values indicate hair that was more tacky or sticky when touched. The average of these results by each evaluator for each of the aerosol hair sprays are summarized in Table I.

TABLE I

Effect of Tg in Aerosol Hair Spray Formulations

5	Subjective Evaluations		
	Evaluator	30°C. Tg Hair Spray	38°C. Tg Hair Spray
	A	4.0	8.5
	B	3.0	9.0
10	C	3.0	8.0
	D	3.5	8.0
	E	4.5	8.5
	F	4.0	8.0
	G	2.5	9.0
15	H	3.0	7.0
	I	4.0	8.0
	<u>J</u>	<u>3.5</u>	<u>9.0</u>
	AVERAGE	3.5	8.3

20

The results in Table I clearly indicate that the aerosol hair spray formulation containing the sulfo-polyester having a Tg of 38°C. proved to be significantly superior in terms of being less tacky or sticky than the hair spray formulation containing the sulfo-polyester having a Tg of 30°C. The average values of 3.5 and 8.3 on the scale of 1 to 10 indicates a significant deviation in tacky or sticky feel to hair treated with the different aerosol hair spray formulations. In addition, the hair spray formulation containing the sulfo-polyester having a Tg of 38°C. had good rinsability/vashability and humidity resistance.

Many variations will suggest themselves to those skilled in this art in light of the above detailed description. All such obvious modifications are within the full intended scope of the appended claims.

WHAT IS CLAIMED IS:

1. An aerosol hair spray formulation comprising:

5 (1) a sulfo-polyester having a glass transition temperature of 36°C. to 40°C. consisting essentially of repeat units from

10 (a) a dicarboxylic acid selected from the group consisting of aromatic dicarboxylic acids, saturated aliphatic dicarboxylic acids, cycloaliphatic dicarboxylic acids, and combinations thereof;

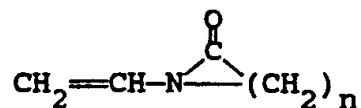
(b) a diol; and

15 (c) a difunctional sulfomonomer containing at least one sulfonate group attached to an aromatic nucleus wherein the functional groups are hydroxy, carboxy or amino, provided the difunctional sulfomonomer is present in an amount from 4 to 25 mole percent based on 100 mole percent
20 dicarboxylic acid and 100 mole percent diol; and

25 (2) a water-soluble, polyvinyl lactam polymer containing at least 50 mole percent of residues of N-vinyl lactams of the formula

25

30



35

wherein n is 3 or 4;

(3) a liquid vehicle consisting essentially of water; and

40 (4) a propellant selected from the group consisting of a C₁- C₄ aliphatic hydrocarbon, dimethyl ether, and mixtures thereof.

2. An aerosol hair spray formulation comprising:

(1) 1 to 10 weight percent based on the weight of components (1), (2), (3) and (4) of a sulfo-polyester having a glass transition temperature of 36°C. to 40°C.

5 consisting essentially of repeat units from

(a) a dicarboxylic acid selected from the group consisting of aromatic dicarboxylic acids, saturated aliphatic dicarboxylic acids, cycloaliphatic dicarboxylic acids, and combinations thereof;

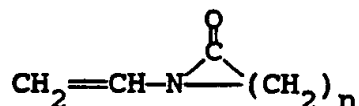
(b) a diol; and

(c) a difunctional sulfomonomer containing at least one sulfonate group attached to an aromatic nucleus wherein the functional groups are hydroxy, carboxy or amino, provided the difunctional sulfomonomer is present in an amount from 4 to 25 mole percent based on 100 mole percent dicarboxylic acid and 100 mole percent diol; and

(2) 1 to 7 weight percent based on the weight of components (1), (2), (3) and (4) of a water-soluble, polyvinyl lactam polymer containing at least 50 mole percent of residues of N-vinyl lactams of the formula

25

30



35

wherein n is 3 or 4;

(3) 46 to 94 weight percent based on the weight of components (1), (2), (3) and (4) of a liquid vehicle consisting essentially of water; and

(4) 3 to 40 weight percent based on the weight of components (1), (2), (3) and (4) of a propellant selected from the group consisting of a $C_1 - C_4$ aliphatic hydrocarbon, dimethyl ether, and mixtures thereof.

3. An aerosol hair spray formulation comprising:

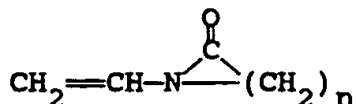
(1) 3 to 7 weight percent based on the weight of components (1), (2), (3) and (4) of a sulfo-polyester having a glass transition temperature of 36°C. to 40°C. consisting essentially of repeat units from

(a) a dicarboxylic acid selected from the group consisting of aromatic dicarboxylic acids, saturated aliphatic dicarboxylic acids, cycloaliphatic dicarboxylic acids, and combinations thereof;

(b) a diol; and

(c) a difunctional sulfomonomer containing at least one sulfonate group attached to an aromatic nucleus wherein the functional groups are hydroxy, carboxy or amino, provided the difunctional sulfomonomer is present in an amount from 4 to 25 mole percent based on 100 mole percent dicarboxylic acid and 100 mole percent diol; and

(2) 1 to 4 weight percent based on the weight of components (1), (2), (3) and (4) of a water-soluble, polyvinyl lactam polymer containing at least 50 mole percent of residues of N-vinyl lactams of the formula



wherein n is 3 or 4;

(3) 55 to 70 weight percent based on the weight of components (1), (2), (3) and (4) of a liquid vehicle consisting essentially of distilled or deionized water; and

(4) 30 to 40 weight percent based on the weight of components (1), (2), (3) and (4) of a dimethyl ether propellant.

10 4. The aerosol hair spray formulation of Claim 1 wherein the dicarboxylic acid component is selected from the group consisting of terephthalic acid, phthalic acid, isophthalic acid, naphthalene-2,6-dicarboxylic acid, cyclohexanedicarboxylic acid, cyclohexanediacetic acid, and mixtures thereof.

15 5. The aerosol hair spray formulation of Claim 4 wherein the dicarboxylic acid component is isophthalic acid.

20 6. The aerosol hair spray formulation of Claim 1 wherein the diol component is selected from the group consisting of ethylene glycol, diethylene glycol, triethylene glycol, 1,4-cyclohexanedimethanol, and mixtures thereof.

25 7. The aerosol hair spray formulation of Claim 6 wherein the diol component is a mixture of diethylene glycol and 1,4-cyclohexanedimethanol.

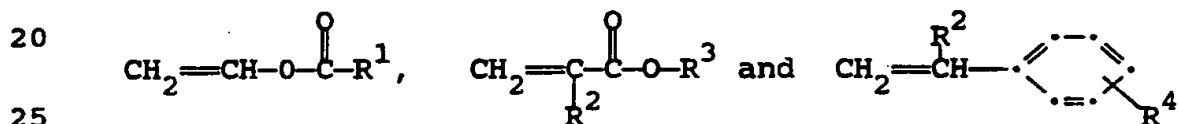
30 8. The aerosol hair spray formulation of Claim 1 wherein the difunctional sulfomonomer component is selected from the group consisting of sulfophthalic acid, sulfoterephthalic acid, sulfoisophthalic acid, 35 4-sulfonaphthalene-2,7-dicarboxylic acid, and esters

thereof.

9. The aerosol hair spray formulation of Claim 8 wherein the difunctional sulfomonomer component is
5 5-sodio-sulfoisophthalic acid.

10. The aerosol hair spray formulation of Claim 1 wherein the sulfo-polyester, component (1), has repeat units from isophthalic acid, diethylene glycol and
10 1,4-cyclohexanedimethanol, and 5-sodio-sulfoisophthalic acid.

11. The aerosol hair spray formulation of Claim I wherein the water-soluble, polyvinyl lactam polymer, component (2), contains up to 50 mole percent of
15 residues of vinyl esters and vinyl aromatic compounds selected from the group consisting of



30 and mixtures thereof, wherein R^1 is alkyl; R^2 is hydrogen or methyl; R^3 is alkyl or alkyl substituted
35 with hydroxy or amino, alkylamino or dialkylamino; and R^4 is hydrogen or alkyl.

40
12. The aerosol hair spray formulation of Claim 1 wherein the alcohol component of the water/alcohol mixture, component (3), is selected from the group consisting of ethanol, isopropanol, t-butanol and
45 combinations thereof.

13. The aerosol hair spray formulation of Claim 1 wherein the liquid vehicle additionally contains 35 to 45 weight percent alcohol.

5 14. The aerosol hair spray formulation of Claim 1 wherein the propellant, component (4), is a
10 C₁ - C₄ aliphatic hydrocarbon selected from the group consisting of methane, ethane, propane, n-butane, isobutane, and combinations thereof.

15 15. The aerosol hair spray formulation of Claim 14 wherein the propellant, component (4), is a mixture containing 83 weight percent isobutane and 17 weight percent propane.

20 16. The aerosol hair spray formulation of Claim 1 which additionally contains an additive selected from the group consisting of preservatives, fragrances, antifoaming agents, hair conditioners and plasticizers.

25

A. CLASSIFICATION OF SUBJECT MATTER IPC 5 A61K7/06 A61K7/00		
According to International Patent Classification (IPC) or to both national classification and IPC		
B. FIELDS SEARCHED Minimum documentation searched (classification system followed by classification symbols) IPC 5 A61K		
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched		
Electronic data base consulted during the international search (name of data base and, where practical, search terms used)		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X,P	EP,A,0 551 748 (UNILEVER) 21 July 1993 see the whole document ---	1-16
X	US,A,5 158 762 (PIERCE) 27 October 1992 see the whole document ---	1-16
X	RESEARCH DISCLOSURE vol. 326 , June 1991 , GB pages 390 - 391 XP206562 'hair grooming compositions' see page 390 - page 391 ---	1-16
Y	US,A,4 300 580 (O'NEILL ET AL.) 17 November 1981 cited in the application see the whole document ---	1-16
-/--		
<input checked="" type="checkbox"/> Further documents are listed in the continuation of box C. <input checked="" type="checkbox"/> Patent family members are listed in annex.		
* Special categories of cited documents : <div style="display: flex; justify-content: space-between;"> <div style="width: 48%;"> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> </div> <div style="width: 48%;"> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"&" document member of the same patent family</p> </div> </div>		
Date of the actual completion of the international search 10 March 1994		Date of mailing of the international search report 28.03.94
Name and mailing address of the ISA European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+ 31-70) 340-2040, Tx. 31 651 epo nl, Fax: (+ 31-70) 340-3016		Authorized officer Fischer, J.P.

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	FR,A,2 300 547 (HOECHST AG) 10 September 1976 see the whole document -----	1-16

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
EP-A-0551748	21-07-93	US-A- 5266308	30-11-93
		AU-A- 3029892	24-06-93
		CA-A- 2085640	21-06-93

US-A-5158762	27-10-92	WO-A- 9317658	16-09-93

US-A-4300580	17-11-81	NONE	

FR-A-2300547	10-09-76	DE-A- 2506461	26-08-76
		AT-B- 351681	10-08-79
		AU-A- 1109276	18-08-77
		BE-A- 838614	16-08-76
		GB-A- 1540862	14-02-79
		JP-A- 51106747	21-09-76
		LU-A- 74346	31-12-76
		NL-A- 7601325	17-08-76
		SE-A- 7601650	16-08-76
